

CLAIMS:

1. An apparatus for concentrating solutions in a vaporising receptacle, said receptacle having a mouth for the removal of vapour, the apparatus comprising:
5 support means for supporting the vaporising receptacle with the mouth of the receptacle facing upwards;
rotation means being operable to rotate the vaporising receptacle thus supported at high speed about a substantially vertical rotation axis;
a vacuum pump to reduce the pressure within the vaporising receptacle;
10 and means for sealing the vaporising receptacle to the apparatus to maintain the reduced pressure.
2. An apparatus according to claim 1 further including means for dispensing a solution to be concentrated into the vaporising receptacle.
- 15 3. An apparatus according to claim 1 or claim 2 further including sensing means to measure the temperature of the solution within the vaporising receptacle; and heating means to apply heat to the solution within the vaporising receptacle.
- 20 4. An apparatus according to claim 3 wherein the sensing means is a non-contact temperature sensor.
5. An apparatus according to claim 3 or claim 4 wherein the heating means includes a hot air heater arranged to direct hot air flow onto the receptacle.
- 25 6. An apparatus according to claim 5 wherein the heating means further includes a diverter for controlling the direction of the hot air flow and in one position directing said flow away from the receptacle.
- 30 7. An apparatus according to any one of the preceding claims, further including a control and regulating unit for controlling or regulating at least one of said rotation means, said vacuum pump, said dispensing means, said sensing means and said heating means.

8. An apparatus according to any one of the preceding claims wherein the rotation means is operable to rotate the vaporising receptacle to speeds at which centrifugal force flattens the solution against the side walls of the receptacle.
- 5 9. An apparatus according to claim 8 wherein the rotation means is operable to rotate the vaporising receptacle at speeds of 2000 rpm or higher.
10. An apparatus according to claim 9 wherein the rotation means is operable to rotate the vaporising receptacle at speeds of 3250 rpm or higher.
- 10 11. An apparatus according to any one of the preceding claims further comprising a vaporising receptacle.
12. An apparatus according to claim 11 wherein the rotational axis passes
15 through the mouth of the vaporising receptacle.
13. An apparatus according to claim 12 wherein the vaporising receptacle is substantially cylindrical.
- 20 14. An apparatus according to any one of claims 11 to 13 wherein the vaporising receptacle is a standard vial.
15. An apparatus according to any one of the preceding claims further comprising means for engaging and disengaging the vaporising receptacle with the apparatus.
- 25 16. An apparatus according to any one of the preceding claims further comprising a level sensing means to detect the level of solution in the vaporising receptacle when the receptacle is not rotating.
- 30 17. An apparatus according to any one of the preceding claims further comprising a condenser.
18. An apparatus according to claim 17 wherein there are two condensers, a first condenser being located between the vaporising receptacle and the vacuum pump
35 and a second condenser being connected to the exhaust of the vacuum pump.

19. An apparatus according to any one of the preceding claims further comprising a sample loop in which the solution to be concentrated is buffered for dispensing into the vaporising receptacle.

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20. An apparatus according to any one of the preceding claims further comprising a solution pump arranged to pump the solution to be concentrated into the vaporising receptacle.

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21. An apparatus according to claim 20 wherein the control unit operates the solution pump to pump the solution to be concentrated into the vaporising receptacle substantially continuously whilst the vaporising receptacle is being rotated.

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22. An apparatus according to claim 21 wherein the means for dispensing the solution includes a nozzle.

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23. An apparatus according to claim 22 wherein the nozzle and the solution pump are chosen such that the solution is dispensed into the vaporising receptacle in a continuous jet.

24. An apparatus according to claim 22 or claim 23 wherein the nozzle and the solution pump are chosen such that there is a pressure difference across the nozzle of at least 1 bar.

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25. An apparatus according to any one of the preceding claims wherein the receptacle has a longitudinal axis about which it is rotationally symmetric and the means for supporting and the means for rotating are arranged such that the longitudinal axis of the receptacle is tilted away from the rotational axis.

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26. An apparatus according to claim 25 wherein the tilt between the rotational axis and the longitudinal axis of the receptacle is between 0 and 6 degrees.

27. An apparatus for producing concentrated solutions or dry solvate including a first apparatus according to any one of the preceding claims and a second apparatus

for performing a precursor process which supplies a solution to be concentrated to said first apparatus.

28. An apparatus according to claim 27 wherein the precursor process is any one
5 of high performance liquid chromatography, purification of organic compounds by flash chromatography, purification of organic compounds by preparative scale supercritical fluid chromatography and synthesis of organic compounds using continuous flow techniques.

10 29. A method of concentrating a solution comprising the steps of:
dispensing said solution into a vaporising receptacle, the receptacle having a mouth for the removal of vapour;
supporting said vaporising receptacle with the mouth facing upwards;
rotating the thus supported vaporising receptacle at high speed about a
15 substantially vertical rotational axis;
reducing the pressure in said vaporising receptacle to evaporate at least a portion of the solvent.

30. A method according to claim 29 further including the step of maintaining the
20 temperature of said vaporising receptacle within a predetermined range.

31. A method according to claim 30 wherein the step of maintaining the temperature includes controlling a hot-air heater which is arranged to direct air flow onto the vaporising receptacle.
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32. A method according to claim 31 wherein the step of maintaining the temperature further includes controlling a diverter mechanism which allows the air flow from the hot-air heater to be directed onto or away from the vaporising receptacle.
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33. A method according to any one of claims 29 to 32 wherein said vaporising receptacle is rotated at a speed sufficient to cause centrifugal force to flatten the solution against the side walls of the receptacle.

34. A method according to claim 33 wherein said vaporising receptacle is rotated at a speed of 2000 rpm or greater.

35. A method according to claim 34 wherein said vaporising receptacle is rotated
5 at a speed of 3250 rpm or greater.

36. A method according to any one of claims 29 to 35 wherein the step of rotating the receptacle at high speed is commenced after the solution has been dispensed into said receptacle.

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37. A method according to any one of claims 29 to 36 wherein the step of rotating the receptacle at high speed is commenced before the solution is dispensed into said receptacle.

15 38. A method according to claim 37 wherein the step of dispensing is performed substantially continuously throughout the concentration process.

39. A method according to claim 38 further comprising the step of controlling either the rate at which the solution is dispensed into the vaporising receptacle, or the
20 rate at which solvent is evaporated in said receptacle, such that a uniform film of solution is maintained over the side walls of the receptacle.

40. A method according to claim 39 wherein the step of controlling includes sensing the temperature of two different portions of the receptacle, a first of said
25 portions being an area of the receptacle proximate to the impact area of a heat source maintaining the temperature of the receptacle, and a second of said portions being an area of the receptacle which is distant from the impact area of said heat source, and adjusting either of said rates according to the rate of change in the difference between the two sensed temperatures.

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41. A method according to any one of claims 38 to 40 further comprising the step of controlling the pressure in the receptacle to prevent phase change from liquid to solid as the solution is dispensed.

42. A method according to any one of claims 38 to 41 wherein the dispensed solution is supplied under pressure.

43. A method according to claim 42 wherein the dispensed solution is supplied at
5 a pressure of at least 4 bar.

44. A method according to any one of claims 38 to 43 wherein the solution is dispensed into the vaporising receptacle through a nozzle.

10 45. A method according to claim 44 wherein the nozzle and flow rate are selected and/or controlled such that there is a pressure difference of at least 1 bar across said nozzle.

46. A method according to any one of claims 29 to 45 further including the step of
15 storing the solution to be concentrated in a sample loop prior to dispensing said solution into said receptacle.

47. A method according to any one of claims 38 to 46 wherein said solution is provided to said receptacle or said sample loop directly from a preceding process.
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48. A method according to claim 47 wherein said preceding process is one of: high performance liquid chromatography; purification of organic compounds by flash chromatography; purification of organic compounds by preparative scale supercritical fluid chromatography; or synthesis of organic compounds by continuous flow
25 techniques.

49. A method according to any one of claims 29 to 48 wherein the step of maintaining the temperature of receptacle includes sensing the temperature of the receptacle with a non-contact temperature sensor.
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50. A method according to any one of claims 29 to 49 wherein the receptacle has a mouth at one end through which the solution is dispensed into the receptacle and evaporated solvent is withdrawn from the receptacle, and the axis of rotation passes through that mouth.
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51. A method according to any one of claims 29 to 50 wherein the receptacle is substantially rotationally symmetric about a longitudinal axis, and that longitudinal axis is tilted away from the rotational axis.

- 5 52. A method according to claim 51 wherein the rotational axis is tilted from the longitudinal axis of the receptacle by between 0 and 6 degrees.